SCOPE: This Standard Operating Procedure governs the analysis of all potable water samples being checked for the presence of aldicarb residues.

SAFETY PRECAUTIONS: Observe all safety practices as described in the company safety manual.

Materials:

Aldicarb, analytical standard, Rhone-Poulenc.
Aldicarb sulfoxide, analytical standard, Rhone-Poulenc.
Aldicarb sulfone, analytical standard, Rhone-Poulenc
Helium, National Welders Supply Company.
2-Mercaptoethanol, 98 %, Aldrich.
Methanol, Burdick and Jackson.
Water, Milli-Q® grade.
O-Phthaldehyde, analytical reagent, Fisher Scientific.
Sodium hydroxide, analytical reagent, Mallinckrodt.
Sodium tetraborate decahydrate, ACS reagent, Aldrich.

APPARATUS:

Two HPLC systems are used for the analysis of aldicarb and its metabolites in potable water.

System 1:

Hewlett-Packard 1090 HPLC.
Schimadzu RF-530 fluorescence detector
Hewlett-Packard 3392A integrator
Column HP C_{18}, 2.1 mm x 10 cm, 5 µm.

Instrumental Parameters:

Mobile phase flow rate 0.8 ml/min (helium sparge)
Column temperature 50 °C
Injection volume 250 µl
Fluorescence detection
Excitation 350 nm
Emission 450 nm

Mobile phase composition:

Time after injection (in minutes)	% methanol (v/v) in water
0.00	3
5.50	3
5.60	25
10.50	25
10.60	80
11.00	80
11.50	3

Typical Retention Times (Minutes)

Aldicarb sulfoxide = 5.15
Aldicarb sulfone = 5.78
Aldicarb = 10.28

System 2:

Hewlett Packard 1090 series 2 HPLC
Hewlett Packard. fluorescence detector
Hewlett Packard 3396B integrator
Column HP C_{18}, 2.1 mm x 10 cm, 5 µm.

Instrumental parameters:

Same as in system 1

Apparatus for post column reaction:

Both systems use Pickering PCX-5000 post column reaction systems.
The flow rate of both solutions (OPA and NaOH) is 0.3 ml/min.

Additional apparatus

Ultrasonic bath
Disposable filter units LC 13 PVDF 0.45 µm (Millipore product # 4452)
Disposable syringes, 3 ml.
Millipore vacuum filteration system
Millipore filter units part number HVLP 047 00

Solutions:

Ortho-phthaldehyde (OPA):

1. Dissolve 5 grams of sodium borate decahydrate in 800 ml of milli-Q water in a 1000 ml Erlenmeyer flask. Stir the solution with a magnetic stirrer.

2. Dissolve 1 gram of phthalic dicarboxaldehyde in 200 ml of methanol. Stir the solution with a magnetic stirrer.

3. Add the phthalic dicarboxaldehyde solution to the sodium borate solution and stir with a magnetic stirrer.

4. Turn the hood on. Add 4 ml of mercaptoethanol to the above solution and stir with magnetic stirrer. This step must be done in the hood.

5. Filter through a millipore filter unit using vacuum filtration system

Sodium Hydroxide:

1. Dissolve 8 grams of sodium hydroxide in 1 liter of Milli-Q® water. Stir the solution with a magnetic stirrer

2. Filter through millipore filter units using vacuum filtration system

Preparation of analytical standards:

Weigh one hundred milligram samples of each of aldicarb, aldicarb sulfoxide and aldicarb sulfone separately into three separate 100 volumetric flasks. Add about 50 ml of milli-Q water to each flask and dissolve the compounds by swirling and bring to volume. Transfer one milliliter of each of these solutions to a 100 milliliter volumetric flask and makeup the volume with water. This is a 10 ppm solution of each compound. Make the appropriate dilutions of this stock solutions to make 1,2,5,20 and 100 ppb standards. Use milli-Q water and grade A glassware for all dilutions. Store all standards in the refrigerator.

Procedure:

1. Filter the water samples through Gelman acrodisc, LC 13 PVDF, 0.45 µm filters using Becton-Dickinson, 3 cc syringes. Samples should be filtered directly into Wheaton 1 ml vials.

2. Cap the vials and load the HPLC sample magazine starting with a 20 ppb standard in position # 0 followed by a 1 ppb standard in position #1 and Milli-Q® water in position # 2.

3. Place standards of 1, 2, 5, 20 and 100 ppb randomly among the samples in the sample magazine with 20 ppb standards in positions 0,12,24,36,48,60,72,84,96.

4. Quantify the residues of aldicarb and its metabolites by the external standard method using 20 ppb standard and peak heights as bases for calculations. The integrator can be calibrated to perform these calculations using 20 ppb standards and the peak heights or peak areas as bases for calibration. Always calibrate from the most recent 20 ppb standard.

   If the instrument does not detect the peak, then the peak heights will be hand measured and the sample concentrations will be calculated using the following formula:

$$\text{Sample Concentration} = \frac{\text{Peak height in sample (mm)}}{\text{Peak height of standard (mm)}} \times \text{Concentration of Standard (ppb)}$$